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Local Microstructure and Stress in Al(Cu) Thin Film Structures Studied by X-Ray Microdiffraction

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ABSTRACT

The microstructure of materials (grain orientation, grain boundaries, grain size distribution, local strain/stress gradients, defects, …) is very important in defining the electromigration resistance of interconnect lines in modern integrated circuits. Recently, techniques have been developed for using submicrometer focused white and monochromatic x-ray beams at synchrotrons to obtain local orientation and strain information within individual grains of thin film materials. In this work, we use the x-ray microdiffraction beam line (7.3.3) at the Advanced Light Source to map the orientation and local stress variations in passivated Al(Cu) test structures (width: 0.7, 4.1 μm) as well as in Al(Cu) blanket films. The temperature effects on microstructure and stress were studied in those same structures by *in-situ* orientation and stress mapping during a temperature cycle between 25°C and 345°C. Results show large local variations in the different stress components which significantly depart from their average values obtained by more conventional techniques, yet the average stresses in both cases agree well. Possible reasons for these variations will be discussed.

INTRODUCTION

Extensive study has been conducted on the mechanical properties of thin films and structures. Thermal expansion mismatch between materials and/or transport of material during electromigration lead to large stresses in these structures, often much higher than those sustainable by bulk materials. Conventional techniques such as wafer curvature and x-ray diffraction only provide a macroscopic average of strain/stress or film texture. As integrated circuit device dimensions shrink to submicrometer sizes, one can expect that local microstructural and stress variations do play a prominent role in determining the materials failure modes. X-ray microdiffraction techniques developed at synchrotron sources [1,2,3] have been shown to be a promising new method in the study of mechanical behavior at the micrometer scale. These techniques allow for the determination of the orientation of single grains in a material as well as the complete strain/stress tensors with micrometer scale resolution. X-rays are particularly advantageous because they are penetrating and can probe buried structures. This means that special sample preparation is unnecessary, allowing the investigation of passivated interconnect structures without altering the stress state.

EXPERIMENTAL

This study was conducted at the x-ray microdiffraction beam line (7.3.3) at the Advanced Light Source. A more detailed description of the beam line is given elsewhere [4]. White x-rays (6 keV – 14 keV) are focused down to a 0.8 x 0.8 micrometer spot using a specially configured Kirkpatrick-Baez mirror pair. Because the beam spot is so small, rotation of the sample is not allowed, as the beam would not stay at the same position on the sample. With the sample fixed we use a white x-ray beam to obtain Laue patterns from individual crystallites. A piezoelectric stage allows for precision positioning of the sample in the focal plane of the x-rays. A 4K x 4K SMART 6000 Bruker CCD collects the Laue pattern at each position on the sample as it is translated. Each CCD frame may contain multiple Laue patterns from different metal grains and the silicon substrate. By translating the sample in this manner, we obtain a series of Laue patterns, which enables us to create maps in stress, strain and orientation.

Automated software allows for rapid processing of the CCD frames. The Si background pattern is digitally removed and the remaining Laue spots are indexed. The software can index overlapping Laue patterns in the same frame and recognize Laue patterns which appear in more than one frame as belonging to the same grain. Using the intensities of the Laue patterns in each frame, a map of the grain structure can be produced. The indexing provides the orientation matrix of each individual grain under the submicrometer illuminated area. The misorientation between any two grains as well as orientation variations within single grains can easily be determined with a precision of 0.01°. The deviations of the Laue spots position from an unstrained crystal are used to calculate the distortion of the crystal unit cell, giving the deviatoric strain tensor with an accuracy of about 2×10^{-4}. Knowledge of the unstrained lattice parameter is unnecessary. The deviatoric stress tensor is simply found by using the anisotropic stiffness coefficients for the material. In this manner, we know the complete orientation and deviatoric stress/strain tensor for each position on the sample.

The samples investigated here are sputtered Al(0.5 wt.% Cu) thin film structures. A 100x100 µm bond pad on the chip (with a thin Ti underlayer) is used to simulate a blanket film (unpassivated, except for the native oxide). The patterned lines have dimensions 0.7 or 4.1 µm in width, 30 µm in length and 0.75 µm in thickness. There are two shunt layers of Ti at the bottom and the top of the lines (thicknesses are 450 Å and 100 Å respectively). The lines are passivated with 0.7 µm of SiO_2 (PETEOS).

The bond pad (blanket film) was thermally cycled between 25°C and 345°C in 40° steps. At each temperature increment, a 15x15 µm area of the film was scanned with the focused white x-ray beam in 1 µm steps. Each scan took approximately 1.5 hours. A 0.7 µm wide line and 4.1 µm wide line were scanned in 0.5 µm intervals at room temperature. In addition, a 0.7 µm line was mapped in 0.5 µm steps across the line and 1 µm steps along the line at several temperatures during a cycle between 25°C and 305°C. The x axis is considered to be along the line, the y across, and the z is out of plane.

RESULTS AND DISCUSSION

Blanket Film

Orientation mapping of the 15x15 µm section of the Al(Cu) bond pad reveals a strong (111) out of plane texture and a random in-plane texture. The out of plane texture ranges from 0 – 3.5° from the sample normal. Grain size ranges from 2.5 μm in diameter to less than 0.5 μm, as revealed by a fine 0.5 μm step scan on a 5x5 μm area of the pad. Due to the limited number of grains illuminated by such small scans, we cannot obtain any accurate statistics on the grain size distribution.

X-ray stress measurement methods which average over a large area assume that the surface of the film is unconstrained and can support no stress ($\sigma_z = 0$)[5]. In order to compare our experimental data, we calculate the average biaxial stress in the film by using the equation:

$$\sigma_{\text{biaxial}} = \frac{\left(\sigma'_x + \sigma'_y\right)}{2} - \sigma'_z$$

where σ'_x , σ'_y , and σ'_z are the average deviatoric stress components in the film. Note that σ'_z is equal in that case to the negative of the hydrostatic stress. A plot of the average biaxial stress in the film obtained by x-ray microdiffraction during a thermal cycle between 25°C and 345°C is shown in Figure 1. This behavior is very similar to that seen by Venkatraman *et al.* [6] who performed wafer curvature measurements on Al(Cu) films, including the stress drop after initial plastic deformation during heating and the increase in slope under 200°C upon cooling.

At the local level σ_z generally cannot considered to be equal to zero, especially near grain boundaries. Our experimental data shows that σ_x' is in general not equal to σ_y', indicating that the stress is only biaxial on average. The complex interaction between neighboring grains induces local triaxial stresses. Because the trace of the deviatoric stress tensor must be zero, the z component is an indication of the variation in the x and y components (in-plane stress). Figure 2 shows a map of σ_z'on the bond pad after completion of the thermal cycle. There are clearly large variations in the local deviatoric stresses.

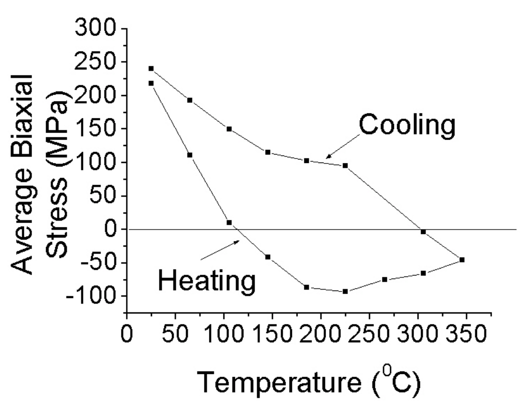

Figure 1: Average biaxial stress vs. temperature for a 15 μm square area of the Al(Cu) film during a thermal cycle.

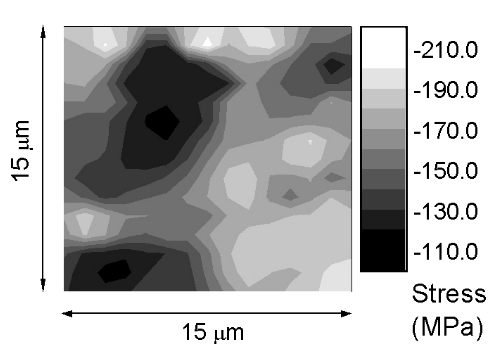

Figure 2: Map of Z component of deviatoric stress for a 15 μm square area of the Al(cu) film at 25°C.

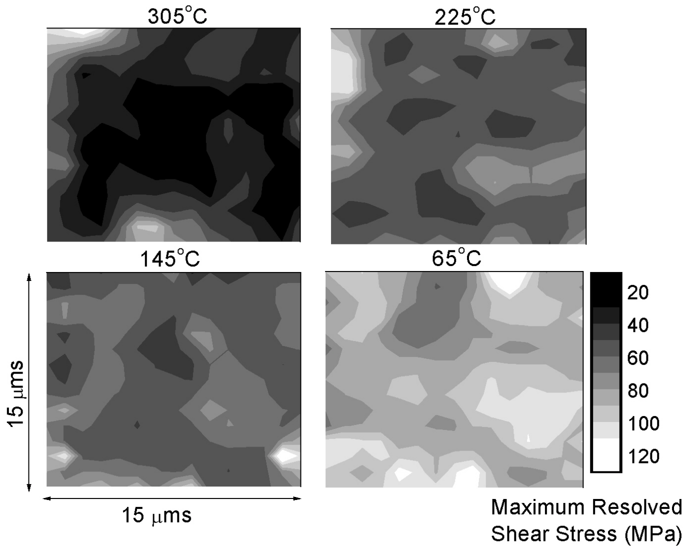

Figure 3: Maximum resolved shear stress for a 15 μm square area of an Al(Cu) film at four temperatures during cooling.

The combination of the deviatoric stress tensor and the complete orientation matrix for a given grain allows us to extract the maximum resolved shear stress (MRSS) for a given slip system. The shear stress is of particular interest when dislocation glide is the dominant deformation mechanism. A map of the resolved shear stress therefore immediately displays where the material is likely to yield first. Figure 3 presents maps of the maximum resolved shear stress on the {111} planes in the <110> type directions (12 independent slip systems) while cooling the film from 305°C to 65°C. The average MRSS at 305°C is 35.5 MPa and increases to 87.5 MPa at 65°C. The average MRSS at 225°C and 145°C are 57.2 and 59.8 MPa, respectively, which means there is very little hardening of the film as it cools over this temperature range. This is clearly visible in Fig. 1. There is a wide range in the MRSS locally at each temperature. Note that these maps cannot be compared with each other directly because each is from a slightly different area on the bond pad, due to sample drift after each temperature change.

<u>4.1 and 0.7 μm Wide Passivated Lines</u>

Grain orientation and stress maps were taken at room temperature for a 4.1 μm and a 0.7 μm wide passivated Al(Cu) line. The grain orientations are similar to those in the Al(Cu) bond pad. While the 4.1 μm line is polycrystalline, the 0.7 μm line appears to be a bamboo-type structure considering the size of the microbeam. In both the 4.1 and 0.7 μm wide lines, we see local variations in the deviatoric stress. In fact, it has been shown in previous work that x-ray microdiffraction is capable of resolving both intergranular and intragranular orientation and stress differences [3]. The stress goes from an average biaxial stress in the pad to a triaxial stress in the narrow line as the level of constraint increases. The average value of the stress along y and z are comparable in the narrowest line. This observation is consistent with an aspect ratio close to one [7,8].

The average deviatoric stress components in the line are plotted in Figure 4 at each temperature during a thermal cycle between 25°C and 305°C. They compare well with those reported by Besser *et al*. [9,10] on similar lines, however, direct comparison cannot be made due to differing passivation materials, processing conditions, and line aspect ratios. The hysteresis in the x and z components indicates some plastic deformation occuring in the line during the thermal cycle. The plastic deformation is greatly reduced compared with the plastic deformation that occurs in the blanket film.

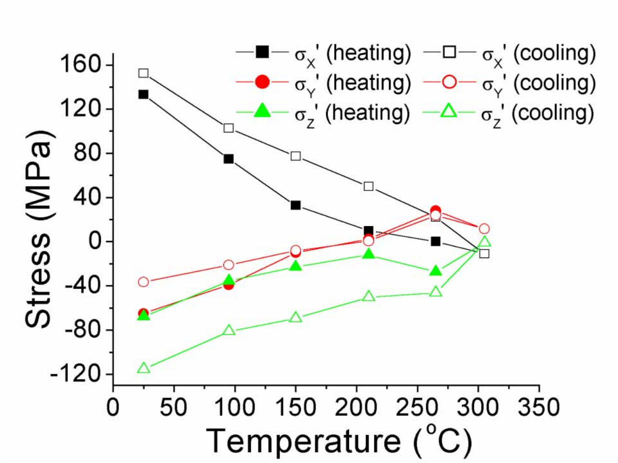

Figure 4: Deviatoric stress components in an Al(Cu) line during a thermal cycle.

This can be attributed to the passivation, which constrains the line on the surface and sidewalls, limiting dislocation motion. The fact that the line is narrow also reduces the average grain size. The effect of passivation constraint on dislocation motion in lines has been described by others [6,11] . Again, the maximum shear stress resolved for the {111}<110> slip system is of particular interest. Figure 5 is a plot of the maximum RSS along the line at three different temperatures when cooling from 150°C to 25°C, with the in-plane and out of plane orientations of the grains along the length of the line. Notice that there are large changes in the shear stress along the line during cooling. The average MRSS increases from 71.2 MPa at 150°C to 112.2 MPa at 25°C as the sample is cooled. By comparison, the average MRSS in the pad at 25°C after the thermal cycle is 95.5 MPa.

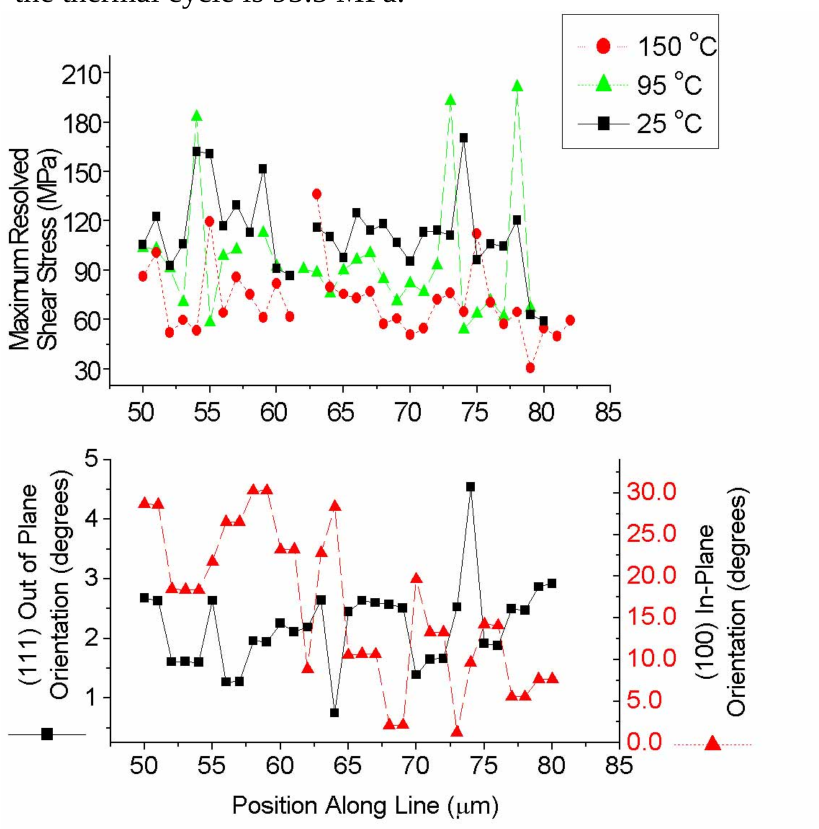

Figure 5: Maximum resolved shear stress and grain orientation along the length of an Al(Cu) passivated line. The shear stress is plotted for three temperatures while cooling the line from 305°C.

The local variations in stress seen in the blanket film and line at a given temperature can be explained by microstructural mechanisms. First, the combination of misorientation between grains and the elastic anisotropy of the crystal lattice will lead to stress variations even if the strain is constant across the grains. This effect is expected to be minimimal due to the (111) texture of the Al(Cu) grains and the small elastic anisotropy of aluminum. The second mechanism is probably due to the grain size distribution.

It is well know that grain boundary strengthening is proportional to the inverse square root of grain size ($g^{-1/2}$) for bulk materials [12]. In polycrystalline thin film materials, the yield stress is proportional to the reciprocal of grain size (g^{-1}) [13,14]. Smaller grains should be able to support higher shear stresses than larger grains, by inhibiting the glide motion of dislocations. Consequently, some grains may yield sooner than their neighbors and lead to a very complex stress state in which high shears are either relieved by yielding or supported until a critical resolved shear stress is achieved. At this time, we have not correlated the shear stress values in a grain at a particular temperature with the grain size.

CONCLUSIONS

X-ray microdiffraction is capable of resolving stress and microstructure at the micrometer scale. We have shown that the local stress state in blanket Al(Cu) films and in passivated Al(Cu) lines is extremely complex. There are large variations in stress between grains, as well as variations within single grains. The stress state of a blanket film can be on average biaxial, but locally the stress is triaxial. Trends in the average data obtained with x-ray microdiffraction agree well with previous studies of stress in thin film structures. Variations in the local stress (on a micrometer scale) can be attributed to a combination of elastic anisotropy, grain misorientations, and grain size effects. Future work will concentrate on following the variation in stress in single grains as a function of misorientation and grain size, as well as interactions with nearest neighbors.

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